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(54) Title: ANTI-CORROSION LUBRICANT COMPOSITIONS AND METHOD OF FORMING JOINT

(57) Abstract

The present invention relates to environmentally advantageous, anti-corrosion lubricating paste compositions comprising a base and a carrier, the carrier being present in a major proportion with respect to the base. Further, in one aspect of the invention, the base comprises wax and graphite, and the carrier comprises petrolatum or mineral oil.

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ANTI-CORROSION LUBRICANT COMPOSITIONS AND METHOD OF FORMING JOINT

Background of the Invention

The present invention relates to environmentally advantageous anti-corrosion lubricating paste compositions comprising a base and a carrier, wherein the carrier is present in a major proportion with respect to the base. Further, the present invention relates to a method of joining oil country tubulers having threading.

In the oil industry, and in other industries, it is necessary to prepare mating surfaces for contact with each other using various types of thread compounds or lubricants. The use of lubricants in preparing pipe connections is well known. Such lubricants reduce friction of components when they are threaded or otherwise engaged. Further, such compositions may be used to inhibit corrosion of metal surfaces.

Lubricant compositions may be used in the oil industry in rotary shoulder connections, drill pipe applications, collars, tool joint connections, and other connections wherein surfaces must be mated together in a tightly bonded, yet releasable condition.

Metal-to-metal contact surfaces are sometimes prepared with lubricants that contain heavy metals. In particular, lead, zinc, and copper have been used in heavy metal-containing lubricants. In such lubricants, it is common to use ordinary grease as a carrier to deliver the lead, zinc, copper, or other heavy metal to the threaded area to be lubricated.

There are numerous problems associated with such heavy metal-containing compositions and greases. For instance, such greases may be harmful to the environment. In both land-based and ocean op rations, the Environmental Protection Agency and the Coast Guard

provide strict limits as to the type and quantity of materials that may be discharged into th environment. Notwithstanding high standards of care in the lubrication of contact surfaces, at least some portion of the greas carrier undoubtedly may be deposited into the environment, in violation of regulations, and to the likely detriment of the environment. Further, such heavy greases cannot easily be removed from threaded connections. When joints must be cleaned for inspection purposes, or when a different type of composition must be applied for storage of the threaded article, the heavy metal-containing lubricants must be removed from the metal surface.

Removal of heavy metal-containing greases from a metal surface typically is accomplished with solvents such as gasoline, kerosene, or some other lightweight hydrocarbon. Such removal frequently results in spillage of solvent into the environment, whether it be on land or into a body of water. Such solvents pollute the environment. Furthermore, to prevent such solvents from being spilled into the environment, they must be collected and disposed of as required under applicable environmental regulations. Such disposal is inconvenient and expensive.

Surprisingly, it has been discovered that a thread composition with performance characteristics comparable to that of heavy metal-containing greases is provided pursuant to the present invention. Such composition is comprised almost entirely of food grade materials that pose very little, if any, threat to the environment in which they are used. The thread composition of the present invention may be used to inhibit corrosion of metal surfaces and to lubricate threaded connections without the disadvantages associated with prior art compositions.

Further, the compositions of the present invention inhibit galling. Steel pipe joints may experience galling, in which damage occurs to the surface of the steel threads where the threads undergo deformation beyond their elastic limit. The compositions of the present invention advantageously limit and greatly inhibit the galling that occurs to steel threads.

Compositions of the present invention also may be applied to steel surfaces that are not threaded to provide corrosion protection and to reduce rust. Such application may be by wiping or spraying upon the steel surface. Further, such application may occur by hot dipping wherein a composition of the present invention is heated above its respective melting point to form a liquid, and the metal or steel surface is dipped into the liquified composition to form a protective barrier to corrosion.

Summary of the Invention

The present invention relates generally to an environmentally advantageous, anti-corrosion lubricating paste composition comprising a base and a carrier, the carrier being present in a major proportion with respect to the base. In the present invention, petrolatum may be used as the carrier, and it may be used in conjunction with mineral oil. Further, the base may be comprised of wax and other inorganic components, including graphite.

In another aspect of the present invention, an environmentally advantageous, anti-corrosion, lubricating paste composition is provided, wherein a carrier comprising petrolatum or mineral oil is present, and such carrier provides the major proportion of the overall composition; and a base comprises wax and inorganic components, wherein such inorganic components comprise no more than about 30% graphite.

In another aspect of the present invention, a joint or threaded connection is provided containing the composition described above.

In yet another aspect of the present invention, the inorganic component of the base further comprises sodium bicarbonate, lithium carbonate, corn starch powder, molybdenum, or graphite.

In another aspect of the present invention, the wax may comprise synthetic wax, natural wax, or both synthetic wax and natural wax.

In another aspect of the present invention, an environmentally advantageous, anti-corrosion, lubricating paste composition is provided. In this aspect of the invention, the carrier comprises food grade petrolatum and mineral oil, and the carrier is also present as a major proportion with respect to the overall composition. The base comprises natural or synthetic wax, including an inorganic component, whereby the inorganic component comprises no more than 30% graphite, and includes sodium bicarbonate, lithium carbonate, corn starch powder, and molybdenum.

In yet another aspect of the present invention, a threaded connection, pipe joint, or drill pipe joint is provided using the composition described above.

In a further aspect of the present invention, a threaded pipe joint is provided comprising a male threaded member and a female threaded member joined to form a threaded engagement, the threaded engagement including void spaces that are substantially filled with a paste composition comprising a petrolatum containing carrier and a graphite containing base, where the paste composition contains no more than about 30% graphite by weight.

In another aspect of the present invention, the above joint is provided wherein th base contains natural

or synthetic wax and one or more of the following: sodium bicarbonate, lithium carbonate, molybd num, corn starch powder, or corn meal.

Description of the Preferred Embodiments

In the preferred embodiments of the present invention, petrolatum is a major component of the carrier composition. The petrolatum preferably is a USP grade (edible) petrolatum produced by Pennzoil Corporation under the trademark PENRECO SNOW. Such petrolatum may be obtained from the Penreco Division of the Pennzoil Corporation.

Graphite is an allotropic form of the element carbon and probably is best characterized by its advantageous thermal stability at temperatures up to about 2600° Kelvin. For purposes of making compositions of the present invention, either synthetic graphite or natural graphite may be used. Graphite consists of layers of hexagonally arranged carbon atoms in a planar condensed ring system. These layers of carbon atoms are substantially flat and are oriented substantially parallel and equidistant to one another. These layers, or basal planes, are linked together and arranged in crystal structures.

The preferred graphite for use in the present invention is 99.9% pure. The preferred graphite is obtained from Southwestern Graphite Company, and is sold under the trade name HPN-325. It is preferable to use a natural flake graphite of 44 micron size.

Several inorganic materials may be used in the carrier of the composition. Sodium bicarbonate (also known as sodium hydrogen carbonate) ($NaHCO_3$) is a white solid formed by passing an excess of carbon dioxide through sodium bicarbonate or hydroxide solution. For purposes of the pres nt inv ntion, sodium bicarbonate

available from grocery stores or other commercial sources may be used. Commercial sodium bicarbonate is available from several sources, including the Arm & Hammer Company.

Lithium carbonate (Li_2CO_3) is provided pursuant to the present invention. It may be obtained from FMC Corporation--Lithium Division.

Further, one of the inorganic constituents that may be present in the composition of the present invention is elemental molybdenum. Such molybdenum may be obtained from Aldrich Chemical Corporation. It is preferably 99.95% pure.

Corn starch powder may be used as an ingredient of the composition of the present invention. Commercial grade corn starch available in grocery stores is sufficient, and a source is Argo brand corn starch available in grocery stores. The corn starch should be 100% USP grade.

As a further component of the present invention, USP grade mineral oil may be used. DRAEKOL 35 mineral oil is preferred. It is believed that DRAEKOL 35 is a trademark of the Penreco Division of Pennzoil Corporation. Such mineral oil may be obtained from the Penreco Division of Pennzoil Corporation.

Several types of natural waxes may be used pursuant to the present invention. In nature, such waxes serve as protective coatings on fruits and leaves, and they are sometimes secreted by insects (for example--beeswax). In general, such natural waxes are a complicated mixture of long-chain alkanes (with an odd number of carbon atoms ranging from C_{25} to C_{35}) and oxygenated derivatives such as secondary alcohols and ketones, as well as esters of long-chain fatty acids and long-chain monohydroxy alcohols. Being highly insoluble in water and having no double bonds in their hydrocarbon chains, natural waxes are chemically inert. It is

believed that suitable vegetable and animal waxes for purposes of the present invention include: beeswax, carnauba wax, Chinese insect wax, Japanese wax, myrtle wax, and spermaceti wax.

Although such waxes, including others, may be used in the present composition, the preferred natural wax is beeswax. Processed 100% pure beeswax may be obtained from several sources, including Dadant Bee Supplies.

Synthetic wax may be utilized in the present invention. The preferred synthetic wax is Witco 666 SUN-O-LITE, a trademark of the Witco Chemical Company. It is an ultraviolet protective corrosion inhibiting synthetic wax.

Further, in certain aspects of the present invention, corn meal may be used. Such corn meal is the typical food grade corn meal that may be obtained from a grocery store. White or yellow corn meal may be used.

If beeswax is used in the present composition, it may be necessary to cleanse the beeswax by filtering it through a 30 US standard sieve filter (0.0234 inch). Such filtering may be accomplished at the time the beeswax is added to the composition by heating the beeswax above its melting point, and then passing it through the filter.

In the process of making the present composition, a relatively small batch may be processed for experimental purposes. The batch size may be increased by increasing the volume of the materials used and the size of the containers.

The invention is further illustrated by the following specific examples:

EXAMPLE 1

Food grade petrolatum (32 ounces by volume) is placed in a final mixing container (container 1) and such

container is heated by an electric surface burn r. The petrolatum is heated to 150°F, plus or minus 10°F, and then mechanically or manually agitated by stirring. After the temperature stabilizes at approximately 150°F, 5.0 grams by weight of powdered graphite is deposited into container 1 and allowed to stabilize for five minutes. The mixture in container 1 is stirred to homogenize the mixture. In the next step, 2.0 grams of lithium carbonate is added to the container 1, and stabilized for five minutes. Next, molybdenum (0.5 grams) is added to container 1, and the mixture is stabilized for five minutes.

Witco 666 synthetic wax is heated in a separate container (container 2) to a temperature of 150°F, plus or minus 10°F. After stabilizing at approximately 150°F, the Witco 666 wax is poured into container 1. The temperature of container 1 is then elevated to 240°F, plus or minus 10°F. During elevation of temperature, th mixture is stirred.

In a third container (container 3), 2.0 ounces by weight of beeswax is heated to a temperature of 200°F, plus or minus 10°F. The temperature is stabilized at about 200°F, and the beeswax is stirred for five minutes. After stabilization, 5.0 grams by weight of sodium bicarbonate is added to container 3, and the mixture is stabilized for an additional five minutes. Next, 5.0 grams by weight of corn starch powder is added to container 3. The mixture is allowed to stabilize for five minutes.

In a fourth container (container 4), 50 milliliters (ml.) by volume of mineral oil is heated to 150°F. The mixture is then added to container 3. Next, the new mixture in container 3 is stabilized for five minutes.

The t mperature of container 3 is then el vated to 250°F, plus or minus 10°F, and stabilized for fiv minutes.

As the next step, the beeswax mixture (container 3) is poured into the final mix container (container 1) containing the petrolatum mixture. The temperature is stabilized at 240°F, plus or minus 10°F.

Container 1 is removed from the heat source, and agitated or stirred from the bottom up with a low speed mechanical mixer. After cooling, or during cooling, the composition may be transferred into containers for storage, or it may be mechanically extruded or pressed into tubes or other convenient packaging means. The composition is air cooled until the product reaches approximately 100°F. The point at which the composition solidifies is in the range of 120°F to 130°F. Mechanical agitation during cooling is very important to prevent solids, in particular graphite and inorganic solids, from settling to the bottom of the mixture. It is preferred that the mixture remain homogenous while cooling.

EXAMPLE 2

An environmentally advantageous, anti-corrosion, lubricating paste composition that is particularly suitable for low temperature operations, for instance, in extremely cold or arctic conditions, is provided pursuant to Example 2. In this example, the above procedure for Example 1 is followed, with the following exceptions. The amount of mineral oil used is increased from 50 ml. to approximately 100 ml.

The mineral oil is used to adjust the viscosity of the composition. A higher mineral oil content is preferable for cold weather applications because it will decrease the resistance to flow, or viscosity, of the final composition. Further, th amount of graphite used

the point just prior to removing the final mixture from the heat source, and after the mixtur has stabilized at 240°F, 4.0 ounces by weight of corn meal (edible grade) is prepared in a separate container (container 5) to which is added a sufficient amount of isopropyl alcohol to liquify the mixture. The isopropyl alcohol kills bacteria present in corn meal. Approximately 50 ml. by volume of isopropyl alcohol is normally required to liquify the corn meal in this manner. The corn meal is soaked for approximately five minutes, and then the corn meal is strained to remove the isopropyl alcohol.

The corn meal of container 5 (after straining) is then added to container 1, and the mixture of container 1 is heated for another five minutes at 240°F to disburse the corn meal and to drive off any residual isopropyl alcohol. At that point, the composition of container 1 is removed from the heat source and cooled as previously described. In the present invention, it is believed that corn meal may assist in filling voids in spaces between fitted joints to provide a composition that exhibits suitable pressure characteristics for non-metal-to-metal sealing connections. The above composition is particularly useful for 8-round type pipe connections.

Threaded pipe joints are constructed using the compositions of the above three examples by applying the composition onto the threads taking care to cover all of the threads on each fitting. The threads are then assembled under sufficient torque to seal the connection.

TEST DATA

Compositions of the present invention have been tested in several ways. First, some compositions have been tested for their anti-galling properties by measuring th make/break characteristics of joints formed from the compositions. Second, some compositions hav

be n t sted for th ir pressure s aling ability by applying internal gas pressure to joints formed from the compositions. Third, the composition of Example 1 was evaluated for its anti-sheen characteristics. Fourth, static toxicity tests have been conducted to examine nontoxicity to mysid shrimp (Mysidopis bahia). Fifth, some of the compositions have been evaluated for frictional characteristics. Sixth, certain compositions have demonstrated anti-corrosive properties in a salt spray (fog) test.

1. GOOD ANTI-GALLING CHARACTERISTICS

An anti-galling test was conducted using the "make/break test". The compositions of Example 1 and Example 2 were tested for their breakout characteristics after the thread compound was applied, and the pipe was torqued under test conditions. The target torque for each makeup was 10,200 Ft-lbs. Nine complete make and breaks were conducted for the composition of Example 1, and five complete make and breaks were conducted using the composition of Example 2 above. The composition of Example 2 was applied to both a 2-7/8 inch 8-round worn sample and a 2-7/8 inch 8-round new sample.

The results of the anti-galling testing were conclusive that the compounds of the present invention provide very good anti-galling characteristics. No galling or thread damage was noted at any time during th testing of the compounds of Example 1 above. With regard to the compounds of Example 2, the only galling noted was on one sample, and that sample was accidentally over-torqued. All other test samples showed no signs of galling or thread damage.

2. GOOD PRESSURE SEALING CHARACTERISTICS

An internal gas pressure test was conducted using standard American Petroleum Institute (API) procedures using 2-7/8 inch 8-round tubing. The composition of Example 3 was applied to the threads and a joint was mad to API recommended torque. The API test pressure of 9700 pounds per square inch was applied internally with nitrogen and held for 14 hours at room temperature. The example was then heated at 350°F for 12 hours, and then nitrogen gas pressure again was applied to 9700 psi while maintaining 350°F for 4 hours. No leaks were noted during the testing.

3. NO VISIBLE SHEEN

A static laboratory sheen test was performed on a thread compound as described above in Example 1. The compound showed no visible "silvery" or "metallic" sheen on a water surface. That is, there was no evidence of iridescence on the water surface by the sample composition of Example 1.

4. MINIMAL EFFECT UPON THE MARINE ENVIRONMENT

Comparative tests were run under testing conditions pursuant to EPA protocol "Drilling Fluids Toxicity Test", Federal Register, Vol. 50, No. 165, Aug. 26, 1985, pp. 34631-34636. The test results show very little, if any, effect upon mysid shrimp (Mysidopis bahia) using the composition of Example 1. The LC_{50} was estimated at greater than 1,000,000 ppm.

5. GOOD LUBRICITY

Frictional tests were performed on the compounds of Example 1 and Example 2 above. The friction factor for the compound of Exampl 1 abov was 0.94. The friction factor for the compound of Example 2 abov was

0.96. The procedure used to determine friction factors was the same procedure under development by the API Committee on Thread Compounds for Rotary Shouldered Connections.

6. GOOD ANTI-CORROSIVE PROPERTIES

Pipe samples made with the compositions of Example 1 and Example 2 above were subjected to 1500-hour salt spray (fog) testing. The duration of the test was 1500 hours. The threads to which the composition was applied showed no change, but the pipe body, which had no composition applied, showed rust. The compositions of the present invention showed good anti-corrosive properties when subjected to salt spray conditions.

The examples and illustrated embodiments above are for illustration purposes only and are not intended to limit the scope of the present invention. It will be recognized that although the invention has been described in considerable detail, variations and modifications can be made from those described without departing from the spirit and scope of the invention.

WHAT IS CLAIMED IS:

CLAIMS

1. An anti-galling, anti-corrosion, lubricating paste composition comprising a base and a carrier, the carrier being present in a major proportion with respect to the base, wherein said carrier comprises petrolatum and mineral oil and wherein said base comprises wax.

2. The composition of claim 1, wherein the base further comprises a largely inorganic component, wherein said largely inorganic component comprises graphite.

3. An anti-galling, anti-corrosion, lubricating paste composition comprising:

(a) a base, wherein said base comprises wax and a largely inorganic component, wherein said largely inorganic component comprises no more than 30% graphite;

(b) a carrier comprising petrolatum or mineral oil;

(c) said carrier being present in a major proportion with respect to said base.

4. A joint containing the composition of claim 3.

5. A threaded connection containing the composition of claim 3.

6. The composition of claim 3 wherein said wax comprises synthetic wax.

7. The composition of claim 3 wherein said largely inorganic component further comprises sodium bicarbonate or lithium carbonate.

8. The compositi n of claim 3 wherein said largely inorganic component further comprises:

(a) sodium bicarbonate;

(b) Lithium carbonate;

(c) corn starch powder;

(d) molybdenum; and

(e) graphite.

9. The composition of claim 3 wherein said wax comprises natural wax and synthetic wax.

10. The composition of claim 3, wherein:

(a) said wax comprises synthetic wax and natural wax;

(b) said largely inorganic component comprises graphite and one or more compound selected from the following group of compounds: sodium bicarbonate, lithium carbonate, and molybdenum; and

(c) said largely inorganic component does not contain more than 30%, by weight, graphite or molybdenum.

11. An anti-galling, anti-corrosion, lubricating paste composition comprising:

(a) a base, wherein said base comprises natural or synthetic wax and a largely inorganic component, said largely inorganic component comprising no more than 30% graphite, sodium bicarbonate, lithium carbonate, corn starch powder and molybdenum;

(b) a carrier comprising food grade petrolatum and mineral oil;

(c) said carrier being present in a major proportion with respect to said base.

12. A threaded connection containing the composition of claim 11.

13. A joint containing the composition of claim 11.

14. A drill pipe joint containing the composition of claim 11.

15. A threaded joint comprising a threaded male end and threaded female end joined to form a threaded junction, the threaded junction including void spaces that are substantially filled with a paste composition comprising a petrolatum-containing carrier and a graphite-containing base, wherein the paste composition contains no more than about 30% graphite by weight.

16. The threaded joint of claim 15 wherein said base contains natural or synthetic wax and one or more of the following: sodium bicarbonate, lithium carbonate, molybdenum, corn starch powder, corn meal.

17. The method of forming a threaded joint of claim 15, comprising:

(a) applying the paste composition to threads; and

(b) joining the male and female ends to form the threaded joint.

18. An anti-galling, anti-corrosion paste composition suitable for protecting and preserving metal surfaces, comprising:

(a) a base, wherein said base comprises wax and a largely inorganic component, wherein said

largely inorganic component comprises sodium bicarbonate;

(b) a carrier comprising petrolatum or mineral oil; and

(c) said carrier being present in a major proportion with respect to said base.

19. The method of treating a metal surface using the composition of claim 17 by directly applying the composition to the metal surface.

20. The method of claim 19, wherein the composition is applied to the metal surface by spraying.

21. The method of claim 19, wherein the composition is applied to the metal surface by hot dipping.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US93/07403

A. CLASSIFICATION F SUBJECT MATTER

IPC(5) :Please See Extra Sheet.
US CL :252/11, 22, 23, 26, 30
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 252/11, 22, 23, 26, 30

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

APS S Wax and petrolatum and graphite and Li or Na bicarbonate.

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US, A, 3,869,393 (Booker) 04 March 1975 See (All).	1-21
X Y	US, A, 3,928,214 (Naka et al) 23 December 1975 see (col. 1, lines 44-53; col. 5, lines 6- end; claims and abstract).	1-6 and 9 1-21
Y	US, A, 3,983,042 (Jain et al) 28 September 1976 See (col. 3, lines 61-66 and 30-38).	7-8, 10-14, 16+18
Y	US, A, 4,148,970 (Mc Intosh et al) 10 April 1979 See (Abstract and col. 1, lines 31-53).	7-8, 10-14, 16 & 18

[X] Further documents are listed in the continuation of Box C. [ ] See patent family annex.

* Special categories of cited documents:

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/US93/07403

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US, A, 4,872,914 (Howard) 10 October 1989 See (Abstract and col. 1, lines 1-59 and col. 5, lines 57- col. 6, lines 1-11).	1-6, 9, 15, 17 and 19-21
Y	US, A, 5,085,700 (Howard) 04 February 1992 See Entire document.	1-21
Y	US, A, Re 33,760 (Howard) 03 December 1991 See entire document.	1-21

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INTE[redacted]TIONAL SEARCH REPORT

[redacted] International application N .
PCT/US93/07403

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim N .
Y	US, A, Re 33, 760 Howard 03 December 1991. See (All).	1-21

Form PCT/ISA/210 (continuation of second sheet)(July 1992)*

INTERNATIONAL SEARCH REPORT

International application No.
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A. CLASSIFICATION OF SUBJECT MATTER:
IPC (5):

C10M 125/02; 125/10; 127/00; 129/00